ANALYTICAL METHODS FOR TRANS FATTY ACID ANALYSIS

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ABSTRACT

The AOAC Technical Division on Reference Materials is in the process of developing a reference material for the analysis of trans fatty acids. If successful, these materials will become available through the AOAC. Two materials have been prepared, a non-hydrogenated soybean oil with approximately 3% trans fatty acids and a hydrogenated soybean oil with approximately 25% trans. In our effort to find the "true" value, these samples were sent to several experienced laboratories for analysis. Data will be presented showing the variability of the data generated by the IR method and the capillary GC method. Limitations of each method will be discussed. Industry activity in Response to Trans Fatty Acids Concerns. The vegetable oil industry is responding to the concern over trans fatty acids in the diet. They are developing new processing procedures to minimize the formation of trans fatty acids during processing. Current processing practices (specifically deodorization and hydrogenation) and their influence on trans fatty acid formation, will be presented. New processing procedures, aimed at the reduction of trans fatty acids in vegetable oils, will be also discussed.

AOAC Technical Division On Reference Materials
***Trans*-Fatty Acid Reference Material**
<u>Sample Preparation</u>

- Two Soybean Oil Samples Were Obtained Containing Approximately 5% and 30% Total Trans
- Samples Were Melted Under Nitrogen
- Mixed
- Packaged In Glass 7 ml Vials
- Stored In Freezer

AOAC Technical Division On Reference Materials
***Trans*-Fatty Acid Reference Material**
<u>Methods Used For Analysis</u>

- AOCS Cd 14c-94 GC Method
- AOCS Cd 14b-93 GC-IR Method
- AOCS Cd 14-61 IR Method

AOAC Technical Division On Reference Material
***Trans*-Fatty Acid Reference Material**
<u>Distribution Of Material</u>

- 15 Labs Volunteered To Do The Analysis by one Or More Of The Methods
- Each Were Asked To Analyze Each Sample In Duplicate On Three Different Days

AOAC Technical Division On Reference Material
***Trans*-Fatty Acid Reference Material**
<u>Laboratory Response</u>

- GC Method: 5
- GC-IR Method: 4
- IR Method: 5

Trans-Fatty Acid Reference Material
GC
Method AOCS Ce 1c-89
Margarine Oil

Method	Lab	N	Mean	Std. Dev.
GC	7	6	26.53	0.563
GC	4	6	24.37	0.258
GC	2	6	23.49	1.404
GC	5	6	25.12	0.180
GC	3	6	24.09	0.190
	All Labs	30	24.72	1.245

Trans-Fatty Acid Reference Material
GC-IR
AOCS Method Cd 14b-93
Margarine Oil

Method	Lab	N	Mean	Std. Dev.
GC-IR	4	6	33.75	0.561
GC-IR	5	6	23.23	0.142
GC-IR	6	6	29.07	0.312
GC-IR	1	6	25.90	0.141
	All Labs	24	27.99	4.014

Trans-Fatty Acid Reference Material
IR
AOCS Method Cd 14-61
Margarine Oil

Method	Lab	N	Mean	Std. Dev.
IR	4	6	32.53	0.427
IR	2	6	26.16	2.471
IR	5	6	22.33	0.143
IR	6	6	27.92	0.307
IR	1	6	26.02	0.117
	All Labs	30	26.99	3.532

Trans-Fatty Acid Reference Material
GC
GC Method AOCS Ce 1c-89
Salad Oil

Method	Lab	N	Mean	Std. Dev.
GC	7	6	3.59	0.045
GC	4	6	2.83	0.143
GC	2	6	2.28	0.169
GC	5	6	2.79	0.044
GC	3	6	3.65	0.087
	All Labs	30	3.03	0.538

Trans-Fatty Acid Reference Material
GC-IR
AOCS Method Cd 14b-93
Salad Oil

Method	Lab	N	Mean	Std. Dev.
GC-IR	4	6	2.81	0.158
GC-IR	5	6	2.85	0.056
GC-IR	6	6	3.55	0.055
GC-IR	3	6	4.82	0.087
	All Labs	24	3.51	0.834

Trans-Fatty Acid Reference Material
IR
AOCS Method Cd 14-61
Salad Oil

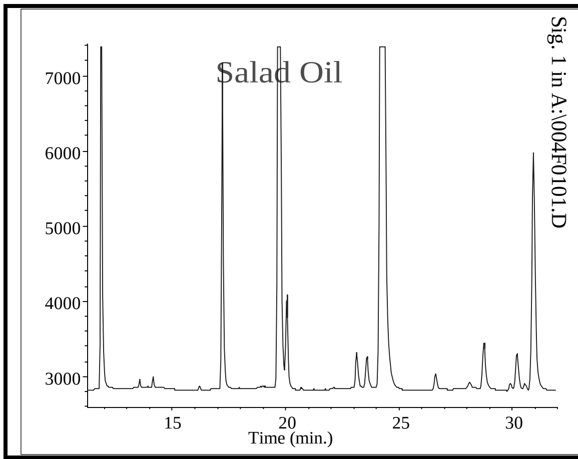
Method	Lab	N	Mean	Std. Dev.
IR	4	6	0.57	0.052
IR	2	6	6.34	0.524
IR	5	6	2.41	0.056
IR	1	6	4.23	0.103
	All Labs	24	3.39	2.200

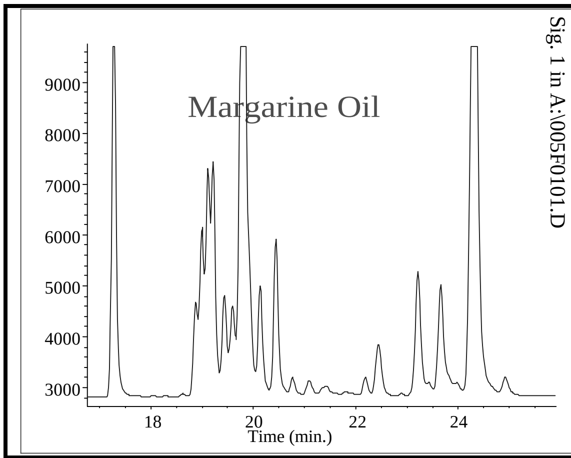
Trans-Fatty Acid Reference Material Total Trans Between Lab Variation Margarine Oil

Method	N	Mean	Std. Dev.
GC	30	24.72	1.245
GC-IR	24	27.99	4.014
IR	30	26.99	3.532

Trans-Fatty Acid Reference Material Total Trans Between Lab Variation Salad Oil

Method	N	Mean	Std. Dev.
GC	30	3.03	0.538
GC-IR	24	3.51	0.834
IR	30	3.39	2.200





GC Method Advantages / Disadvantages

- Advantages
  - Most oil refineries have GC's
  - Simple sample preparation
  - Capable of measuring individual fatty acids
  - Useful for a wide range of sample types
- Disadvantages
  - Difficult interpretation of data
  - Long analysis time

GC-IR Method Advantages / Disadvantages

- This method is only intended for the determination of trans-octadecenoates

GC-IR Method Advantages / Disadvantages

- Advantages
  - Fast
  - Simple sample preparation
- Disadvantages
  - Uses carbon disulfide
  - Data reduction difficult
  - Not applicable to samples with < 5% total *trans*

Project Status

- GC method appears to be the method of choice
- Resolution factor of 1 for *trans*-13 and oleic will be a recommended change in method
- A recognized expert, Dr. Ratnayake, Health Canada has re-analyzed the samples
- An expert review committee of six is reviewing the data and will comment on where we should go from here

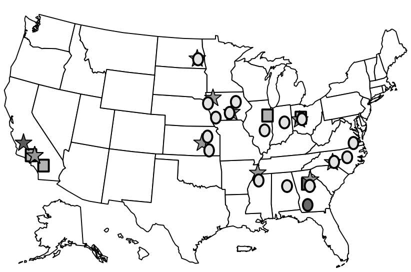
Why Use Reference Materials ?

- Get everyone on the same page
- Help everyone understand the variability of methods used
- Use with SPC techniques (pre-control) to control the capability of methods

Cargill Worldwide Oilseeds Processing

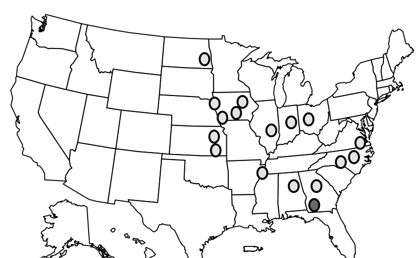
▼United States	▼United Kingdom	▼Australia
▼Canada	▼France	▼Malaysia
▼Venezuela	▼Netherlands	▼Phillipines
▼Brazil	▼Belgium	▼China
▼Argentina	▼Spain	

U.S. Oilseeds Processing - Physical Assets



Crushing Facility
Refining Facility
Packaging Facility

U.S. Oilseeds Processing - Crushing Facilities

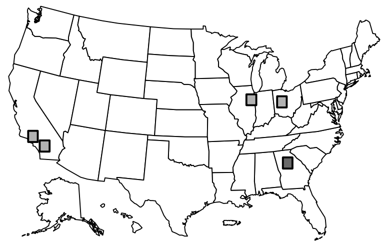


Bloomington, IL	Guntersville, AL	Norfolk, VA
Cedar Rapids ,IA	Iowa Falls, IA	Raleigh, NC
Dawson, GA	Kansas City, KS	Sidney, OH
Des Moines, IA	Lafeyette, IN	Sioux City, IA
Fayetteville, NC	Memphis, TN	West Fargo, ND
Gainesville, GA		Wichita, KS

U.S. Oilseeds Processing - Refining Facilities

Des Moines, IA	Gainesville, GA	Sioux City, IA
Fayetteville, NC	Memphis, TN	West Fargo, ND
Fullerton, CA	Sidney, OH	Wichita, KS
		Vernon, CA

U.S. Oilseeds Processing - Packaging Facilities

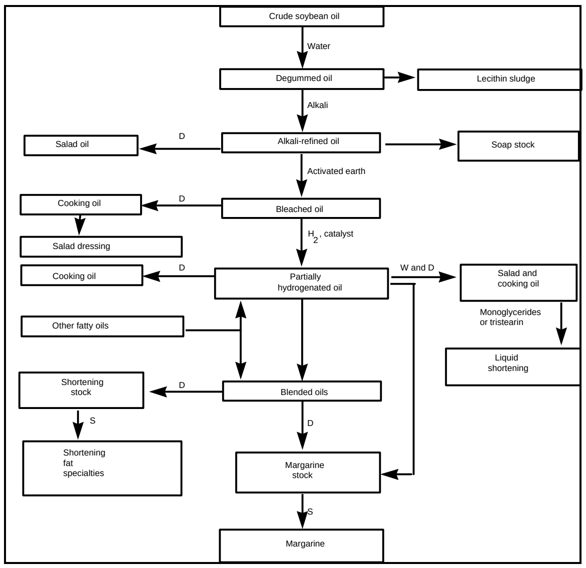


Chicago, Il	Gainesville, GA	Sidney, OH
Fullerton, CA	Vernon, CA	

Raw Materials Processed

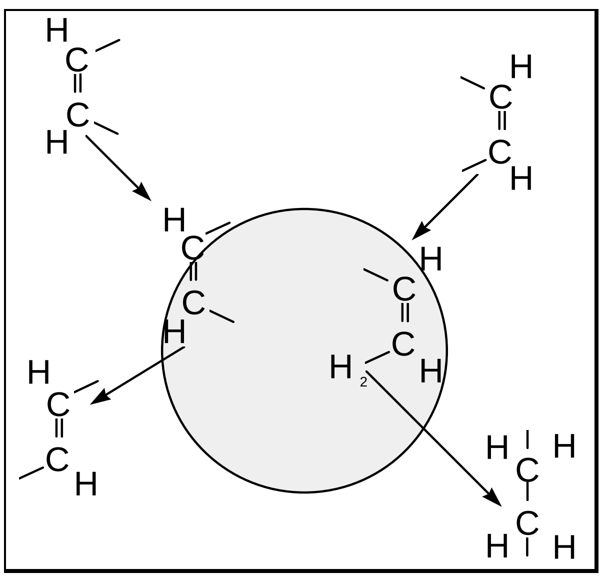
Protein
- Canola
- Corn
- Peanut
- Soybean
- Sunflower
- Specialty Crops
  - canola
  - soybean
  - sunflower

Oil
- Corn
- Cottonseed
- Palm
- Peanut
- Soybean
- Sunflower
- Specialty Oils
  - canola
  - sunflower
- Tallow



Hydrogenation

- In the presence of a catalyst, hydrogen gas is added to the double bonds of fatty acids
- Changes the melting behavior of oils, converting oils to semisolids
- Reduces the iodine value
- Improves oxidative stability
- Isomeric (*trans*) unsaturated fatty acids formed



Industry Activity
Development of Low Trans Products

- Optimization of current processes
  - Time exposed to high temperature
  - Lower temperature during hydrogenation
  - Hydrogen pressure
  - Catalyst recycling
- Development of new processes
  - Time
  - Temperature
  - Precious metal catalysts

Deodorization

- High temperature, high vacuum, steam distillation
- Can produce isomeration, both positional and geometric
- Removes
  - Flavors and odors
  - Free fatty acids
  - Sterols
  - Hydrocarbons
  - Pigments

Deodorization

- Minimization of isomerization by optimizing
  - Temperature
  - Time
  - Vacuum
  - Stripping steam rate

Other Industry Activities
Production of Healthy Fats and Oils

- Plant breeding
  - High Oleic Sunflower
  - High Oleic Soy
  - Canola with unique properties
- Interesterification
  - Moving fatty acids to different positions in the triglycerides in the fat
    - Random - chemical catalysis
    - Selective - enzyme catalyzed
  - Changes the melting properties of fats and oils
  - Example: Interesterification of high melting fats with oils to produce products with a variety of melting properties